

TABLE 1

Example	Solids content [%] <sup>a</sup>	K value <sup>b</sup>	pH (tq)	Mw <sup>c</sup>	PDI <sup>c</sup>	Oligomer content <1000 g/mol	P % internal <sup>d</sup>	P % external <sup>d</sup>	P % inorg <sup>d</sup>
1	42.5	24.8	4.3	5080	2.1	4.7	79.6	11.3	9.2
2	41.5	24.9	4.3	4990	2.1	4.9	81.6	6.9	10.5
3	42.1	24.1	4.3	4820	2.0	5.2	85.8	6.4	7.8
4	43.6	23.2	4.5	4960	2.1	5.4	86.7	5.6	7.7
5	41.6	26.0	4.3	5490	2.1	4.4	65.1	13.0	20.5
6	46.4	16.6	4.2	3040	1.6	6.4	86.3	8.1	5.6
7	45.8	30.3	4.2	8020	2.4	2.5	80.8	11.7	7.5
8	46.1	24.0	4.3	4990	1.9	3.4	83.9	10.2	5.9
9	43.5	23.7	4.3	5080	2.0	3.8	86.7	5.6	7.7
10	58.6	23.5	1.8	4610	1.8	3.7	75.9	18.8	5.3

<sup>a</sup>ISO 3251, (0.25 g, 150° C., 2 h)<sup>b</sup>determined by Fikentscher method with 1% solution in completely ion free water<sup>c</sup>determined by gel permeation chromatography<sup>d</sup>determined with <sup>31</sup>P{<sup>1</sup>H} and <sup>31</sup>P NMR

## Performance Tests

## Use of Acrylic Acid Polymers as Dispersants

The polyacrylic acid solutions obtained were tested for their usefulness as dispersants for producing slurries. For this, calcium carbonate was in each case ground using a Dispermat. For this, in each case, 300 g of calcium carbonate (Hydrocarb OG from Omya) and 600 g of ceramic beads were mixed and initially charged to a 500 ml double-wall vessel filled with tap water. Then, 100 g of a 3% by weight aqueous solution of the in-test polyacrylic acid was added after adjustment to pH 5.0. The grinding was done using a grinding assembly of the type Dispermat AE-C (from VMA-Getzmann) with a cross-beam stirrer at 1200 rpm. As soon as 70% of the pigment had a particle size (PSD) of less than 1 μm, the grinding operation was terminated (about 70 min, LS 13320 particle measuring instrument from Beckman Coulter). After grinding, the slurry was filtered through a 780 μm filter using a porcelain suction filter to remove the ceramic beads, and the solids content of the slurry was adjusted to 77%. The viscosity of the slurry was determined at once, after 24 h and after 168 h using a Brookfield DV II viscometer (using spindle No. 3).

The results of the dispersing tests are summarized in table 2.

TABLE 2

Example	Particle size		Dynamic viscosity [mPas] at 100 rpm				Slurry solids content [%]
	distribution		after	after	After	after	
	<2 μm	<1 μm	1 h	24 h	96 h	168 h	
1	99.1	74.0	527	930	1750	2450	77.0
2	98.9	72.9	620	1870	2220	3341	77.0
3	97.6	72.6	687	1710	2747	3419	77.0
4	97.2	71.1	619	1620	2357	3289	77.0
5	98.9	72.5	820	2540	3960	5270	77.0
6	99.5	74.0	2034	4055	>6000	>6000	77.0
7	99.0	74.0	835	1902	3209	4050	77.0
8	99.1	74.6	524	949	1974	2567	77.0
9	98.9	75.0	628	1448	2280	2890	77.0
10	98.9	72.4	1284	3011	4380	5645	77.0

We claim:

1. A process for preparing aqueous solutions of acrylic acid polymers by polymerization of acrylic acid in feed operation with a free-radical initiator in the presence of hypophosphite in water as solvent, which process comprises

- initially charging water and optionally one or more ethylenically unsaturated comonomers,
- continuously adding acrylic acid in acidic, unneutralized form, optionally one or more ethylenically unsaturated comonomers, aqueous free-radical initiator solution and aqueous hypophosphite solution,
- adding a base to the aqueous solution on completion of the acrylic acid feed,

wherein the comonomer content does not exceed 30% by weight, based on the total monomer content, wherein the aqueous hypophosphite solution is added during a total feed time made up of three consecutive feed time spans  $\Delta t_I$ ,  $\Delta t_{II}$  and  $\Delta t_{III}$ , wherein the average feed rate in the second feed time span  $\Delta t_{II}$  is greater than the average feed rates in the first and third feed time spans  $\Delta t_I$  and  $\Delta t_{III}$ .

2. The process according to claim 1 wherein the first feed time span  $\Delta t_I$  amounts to 30 to 70% of the total feed time.

3. The process according to claim 1 wherein the second feed time span amounts to 5 to 25% of the total feed time.

4. The process according to claim 1 wherein the third feed time span comprises two subsidiary feed time spans  $\Delta t_{IIIa}$  and  $\Delta t_{IIIb}$ , wherein the average feed rate during the first subsidiary feed time span  $\Delta t_{IIIa}$  is not less than the average feed rate during the first feed time span and the average feed rate during the second subsidiary feed time span  $\Delta t_{IIIb}$  is less than the average feed rate during the first feed time span.

5. The process according to claim 1 wherein the total feed time is in the range from 80 to 500 min.

6. The process according to claim 1 wherein up to 30% by weight of comonomers selected from the group consisting of methacrylic acid, maleic acid, maleic anhydride, vinylsulfonic acid, allylsulfonic acid and 2-acrylamido-2-methylpropanesulfonic acid is co-interpolymerized.

7. The process according to claim 1 wherein the polymerization is carried out under inert gas atmosphere.

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